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A Wireless Passive Humidity Threshold Monitoring Solution Based on a Permanent Resistance Change

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Abstract

A wireless passive sensor solution is presented, which permits continuous monitoring of a humidity threshold violation, but does not require a permanent supply of electric energy. It is based on an inkjet printed resistance changing element, which incorporates three mechanisms: the deliquescent behavior of salts, transport processes in porous media and chemical sintering of silver nanoparticles under ambient temperature conditions. Upon exceeding a salt specific deliquescence humidity a salt solution forms in a reservoir (humidity threshold activation), which is transported through a porous substrate (delay mechanism) into a silver nanoparticle region, triggering a chemical sintering under ambient temperature conditions (permanent state change). In dependence of the design and manufacturing parameters comparatively large permanent resistance changes (i.e., 1MΩ and above) can be realized. These humidity triggered switches can be integrated into inductively coupled resonant (ICR) sensor tags, in the presented case, a double planar sensor coil arrangement.

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Keywords: Wireless; Passive; Humidity; Irreversible; Inductively; Coupled; Nanoparticle; Sintering; Chemical;

1. Introduction

Due to the pervasive nature of water, water vapor and moisture have strong influence on the product quality of various goods (e.g., eatables, chemicals, electronics, etc.) and their associated manufacturing, transport and storage processes. Humidity is therefore of strong interest for measurement and control. Full life cycle item identification with standardized, conventional RFID tags is reality, and integration of additional sensor functionality has been demonstrated [1]. Continuous monitoring of environmental parameters, without permanent supply of electric energy (either an omnipresent reader station or an integrated source of electric energy, i.e. battery or harvester) is challenging. Sensor solutions, based on irreversible state changes, suitable for lowest cost manufacturing processes, are a promising solution approach for environmental parameter monitoring on item level, especially for upcoming transistor-less RFID tags. In difference to a previously presented solution [2] based on a freely programmable RIFD tag, in this work an inductively

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coupled resonant sensor (ICR) arrangement with a connected humidity sensitive element is presented. The detection of the exceedance of a r.H. threshold, without the need of a permanent supply of electric energy, is possible by evaluating permanent changes in the frequency response characteristics of the sensor resonator.

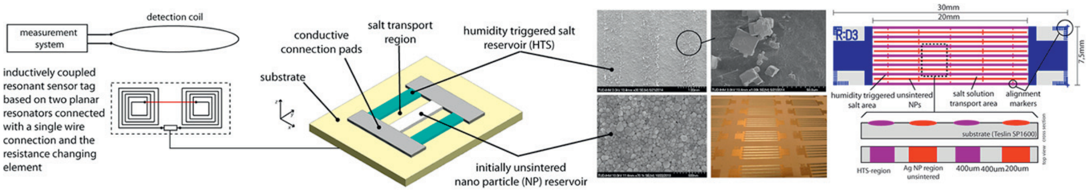
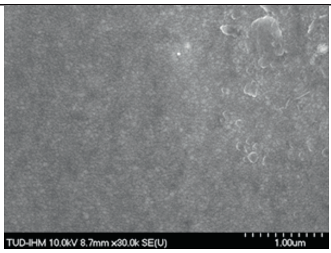
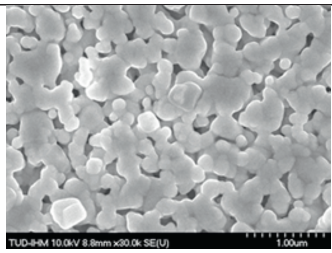
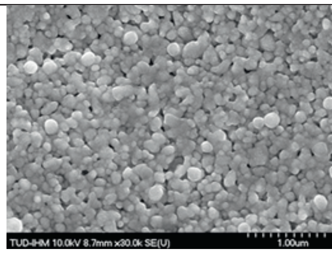


Fig. 1: Inductively coupled resonant sensor incorporating the humidity sensitive element.

2. Sensor principle and humidity sensitive element

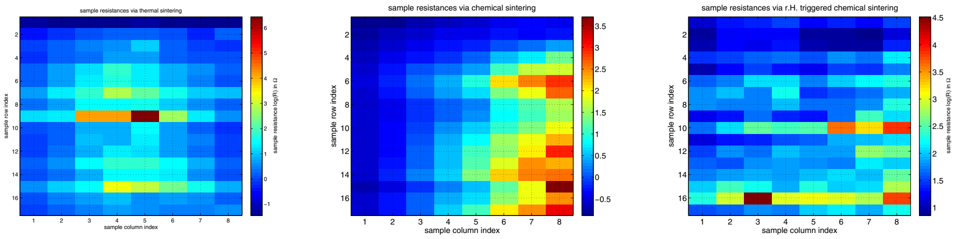
An essential component of the proposed sensor tag is the humidity sensitive element. It's principle exploits the fact that most of the inorganic and several organic salts show a deliquescent behavior [3], in which sorbed water vapor from the surrounding air causes a solid to liquid phase change, which is accompanied with a well defined relative humidity value (DRH). In this work sodium chloride (NaCl) with a DRH of 75.5% at 20°C is used, which temperature dependent DRH variation stays below 1.5% in a range from 5°C to 50°C[4]. Upon DRH exceedance complex porous media transport phenomena (e.g., surface diffusion, capillary transport, ...) cause a spread of water and dissolved ions from the initial reservoir into the nanoparticle region (containing core shell Ag NPs, type XJET JET Solar FF20141). Here a chemical liquid phase sintering process [5] is initiated, leading to a removal of the isolating, protective ligand and the development of an electric percolation path. NPs rapidly become conductive, form larger agglomerates and sinter necks can devevelop (Tab.1) between neighboring particles.

Table 1: SEM images of pre and post sintered (r.H. triggered at 25°C) Ag NPs of the sinter material depot.

unsintered NP $R > 100\,\mathrm{M\Omega}$	r.H. 98% (5h) $R = 4.8\Omega$	r.H. 80%(5h) $R = 24.5\Omega$
		

Humidity sensitive elements are convieniently manufactured with additive, printing technlogies for electronics, exemplified by inkjet printing with a Fujifilm Dimatix DMP-2800 printer. Demonstrators are manufactured on Teslin SP1600 (PPG Industry), a synthetic paper not showing an early substrate facilitated NP sintering. Printing is carried out in a three step process (typically 17x8=136 samples). Used are cartridges with a nominal drop volume of 10 pl and a printing resolution of 1270 dpi. In the first step all NP areas are printed. Afterwards initially conductive regions are chemically sintered (i.e., connection pads) by applying two layers of NaCl solution (0.5 $\frac{\mathrm{mol}}{\mathrm{l}}$). Hereby a manual cartridge replacement is necessary, which currently determines the degree of miniaturization (a min. transport region zone width of 200 μm has been reached). The salt reservoir region is formed by 10 layers of NaCl solution (0.5 $\frac{\mathrm{mol}}{\mathrm{l}}$) and manufacturing follows a storage period of at least 24 h.

Fig.2 shows manufacturing process dependent variations manifested in the final state resistance of humidity sensitive elements, made conductive with different sintering strategies (thermal, chemical sintering,



(a) thermal sint. $R_{avg} = 8.2\,\Omega$, $R_{max} = 623.6\,\Omega$, $R_{min} = 0.2\,\Omega$ (b) chemical sint. $R_{avg} = 3.3\,\Omega$, $R_{max} = 40.8\,\Omega$, $R_{min} = 0.4\,\Omega$ (c) r.H. induced sint. $R_{avg} = 8.2\,\Omega$, $R_{max} = 91.5\,\Omega$, $R_{min} = 2.3\,\Omega$

Fig. 2: Comparison of sensor sample resistance values (representing the final sensor state, logarithmic) after applying different sintering mechanisms: a) thermal (135°C,2d) b) chemical (2 layers NaCl of $0.5\frac{mol}{l}$) and c) relative humidity triggered chemical sintering (climate chamber 93%,25°C,1d). Samples are printed row wise (start from 1:1).

humidity triggered chemical sintering). Although possible, an all printed sensor tag was not realized, due to pretests showing small quality factors of printed resonators. For experiments a hybrid strategy of PCB copper resonators connected to humidity sensitive elements has been used.

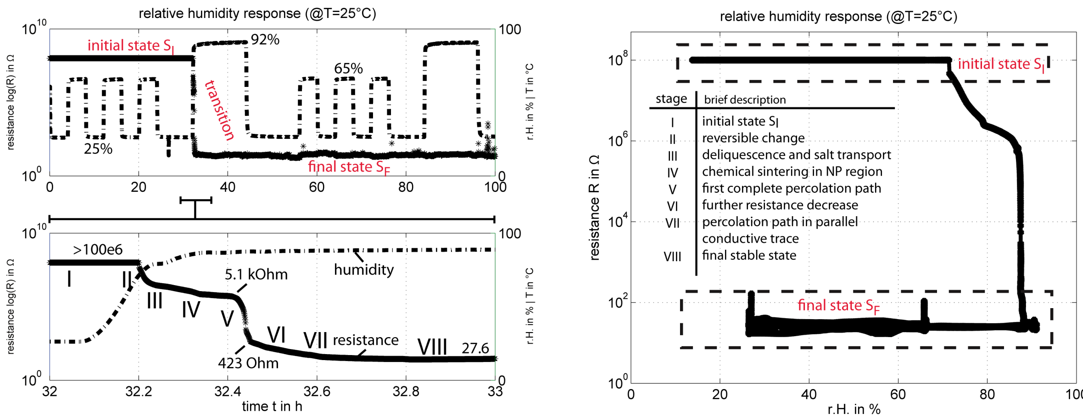
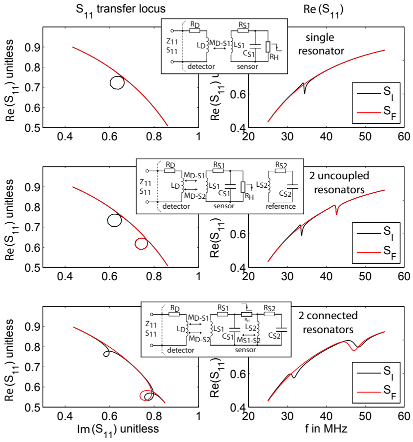


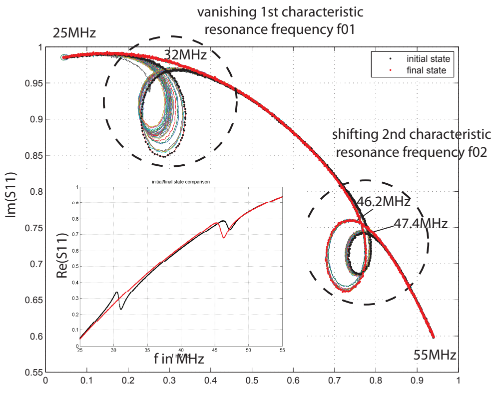
Fig. 3: A representative response of the humidity sensitive element (left), showing the resistance change $R(t, \varphi)$ due to a relative humidity φ above the threshold (top) determined by the DRH of the chosen salt in the salt reservoir.

3. Inductively coupled resonant sensor tag

Simply connecting humidity sensitive elements to the terminals of a spiral resonator leads to a short circuited interwinding capacitance in the final sensor state and therefore a remotely undetectable resonance frequency due to the large resistance changes (Fig.3) of the humidity sensitive element. As shown with SPICE simulations, two connected resonators are advantageous (Fig.4a). Measurement results in Fig.4b demonstrate the feasibility of the solution. The monitored S_{11} parameter (locus curve between 25 to 50 MHz) of a detection coil with coupled sensor tag initially shows two resonances (black curve) resulting from the transformed impedance of the inductively coupled sensor tag (distance 1cm). As a result of a humidity exposure above the DRH, a vanishing first resonance (at $f_{0,1} \approx 32$ MHz) and a shifting second resonance is observed ($\Delta f_{0,2} \approx 1.2$ MHz), which is in qualitative agreement with the simulations. These characteristic changes in



(a) Comparative simulations.



(b) Experimental results.

Fig. 4: Simulations (a) of ICR systems (locus and real part of S_{11} parameter, with initial S_I and final S_F tag states) for different possibilities of connecting the humidity sensitive element: top - the problematic case, with a short circuited sensor resonator in S_F; middle - two unconnected resonators, with vanishing first ($f_{0,1}$) and lasting second resonance ($f_{0,2}$); bottom - proposed case, with two connected resonators leading to a vanishing $f_{0,1}$ and shifted $f_{0,2}$. The measurements (b) prove the feasibility of the approach.

the frequency response are permanent, and they indicate a critical relative humidity exposure of the sensor tag, as well as any good the sensor tag is equipped to.

4. Conclusion

The feasibility of the wireless passive sensor solution has been demonstrated. The exceedance of an upper humidity threshold can be detected after its occurrence by a lasting ICR tag parameter change, manifested in two changes in the tags frequency response characteristic. Due to the simple buildup, the presented sensor concept is a suitable option for printed, lowest cost mass production technologies and shows potential for an application on humidity sensitive goods or an integration in their enclosing wrappings.

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